

Evaluation of the effects of thermal changes on the bond strength between zirconia framework and veneering ceramic during the firing process

Purpose

The aim of this in-vitro study was to evaluate the effect of thermal changes to shear bond strength during the firing process of veneering porcelain on a zirconia framework.

Materials and Methods

Single yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) framework ceramic (Kavo Dental GmbH) and three different types of veneering ceramics (IPS e.max Ceram, Vita VM9, and GC Initial Zr-FS) were used. One-hundred-twenty standard disc-shaped samples were prepared from zirconia blocks by using a CAD/CAM system (Kavo Everest). Four different cooling processes (maximum, 25°C/min, 50°C/min and 75°C/min) were applied to the veneering ceramics and the shear bond strength (SBS) test was performed. Ceramic surfaces were investigated by using scanning electron microscope (SEM). The possible occurrence of a t-m transformation of zirconia was evaluated by X-Ray Diffraction (XRD). Two-way analysis of variance, Bonferroni correction and paired comparisons were used for statistical analysis.

Results

The main effects of veneering ceramics on shear bond strength were found to be significant ($p=0.042$). The mean shear bond strength values differ according to the cooling process ($p<0.001$). The monoclinic phase ratio increased in groups with fast cooling process.

Conclusion

The thermal changes during the firing process of veneering porcelain on a zirconia framework influenced the shear bond strength of the all-ceramic bilayered system. A slow cooling process provided higher strength for bilayer ceramic samples.

Keywords: Zirconia, Veneering ceramic, CAD/CAM, Bond strength, Cooling process

Introduction

The increase in the aesthetic expectations and technological developments have created an area of use for different tooth-colored, biocompatible, high-strength restorative systems as an alternative to metal-supported ceramic restorations that have been used for a long time (1, 2). However, their fragility and low resistance to stress limit the clinical applications of these materials. The use of all-ceramic systems was mostly limited to the anterior region but thanks to the advanced dental ceramics, their use in posterior teeth has also increased. Zirconia, which exhibits high mechanical performance, durability, stress resistance, chemical and dimensional stability compared to other all-ceramic systems, has increased the reliability of all-ceramic systems (3-5).

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In order to obtain a natural appearance, the feldspathic porcelain or various veneering ceramics specially developed for zirconia are applied over the opaque zirconia framework (6). The thermal processes applied during the production and veneering ceramic application affect the mechanical properties of the restoration. In addition, the weakness of veneering ceramics can negatively affect the clinical success of the restoration and may result in clinical failure in the form of fracture (chipping) formation in the veneering ceramics (7). This failure could be related to the difference in the thermal expansion coefficient between framework and veneering ceramics, thickness of the veneering ceramic, elasticity modulus, defective areas that may occur in the porcelain, improper firing process and the thermal changes in the cooling process after firing (8,9).

Residual stresses occur on the surface as a result of the cooling process after firing of the veneering ceramics (10). The authors argued that residual stresses could be changed using different heat treatments and these changes occur as a result of the viscoelastic behavior of the glass as a result of various cooling processes. No clear explanation has been provided so far for bonding problems in zirconia-based ceramics that result in chipping which is most likely caused by the cooling process (8,11). The aim of this in-vitro study was to evaluate the effects of thermal changes on the bond strength between the zirconia framework and the veneering ceramic during the firing process. The null hypothesis tested in the present study is that the thermal changes during the firing process does not affect the bond strength.

Materials and Methods

Sample size estimation

Using the 95% confidence (1- α), 80% test power (1- β), Partial eta squared=0.079308 and $f=0.293$ input, the minimum number of samples to be included was determined as 115 (12).

Table 1. Cooling process and groups of the veneering ceramics

IPS e.max Ceram n=40	Kavo Everest ZS n=120	GC Initial Zr-FS n=40
	Vita VM9 n=40	
Max n=10 (IPS-Max)	Max n=10 (Vita-Max)	Max n=10 (GC-Max)
75°C/min n=10 (IPS-75)	75°C/min n=10 (Vita-75)	75°C/min n=10 (GC-75)
50°C/min n=10 (IPS-50)	50°C/min n=10 (Vita-50)	50°C/min n=10 (GC-50)
25°C/min n=10 (IPS-25)	25°C/min n=10 (Vita-25)	25°C/min n=10 (GC-25)

Table 2. Technical data on the veneering ceramics

Veneering Ceramic	Coefficient of Thermal Expansion (CTE)	Glass Transition Temperature	Chemical Solubility	Flexural Strength	First Dentine Firing
IPS e.max Ceram	9,5±0,2510 ⁻⁶ K ⁻¹	490±10°C	15±5 µg/cm ³	90±10 MPa	750°C
Vita VM9	8,8-9,2 x 10 ⁻⁶ K ⁻¹	600°C	10 µg/cm ³	100 MPa	910°C
GC Initial Zr-FS	9,4 x 10 ⁻⁶ K ⁻¹	550 °C	12 µg/cm ³	90 MPa	810°C

Preparation of bilayered zirconia-veneering ceramic samples

The present study was performed using single yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) framework ceramic (Kavo Dental GmbH, Biberach, Germany) and three different types of veneering ceramics (IPS e.max Ceram, Ivoclar Vivadent, Schaan, Liechtenstein; Vita VM9, Vita Zahnfabrik, Germany; and GC Initial Zr-FS, GC Europe N.V., Interleuvenlaan, Leuven, Belgium) recommended for veneering the zirconia framework (Table 1, Table 2, Table 3).

One-hundred-twenty standard disc-shaped samples (height of 3mm and diameter of 10 mm) were prepared from zirconia blocks by using a CAD/CAM system (Kavo Everest). All samples were airborne particle abraded with 50 mm Al₂O₃ at 2.5 bars of pressure (15 s) at a maximum distance of 10 mm and ultrasonically cleaned in distilled water for 3 minutes followed by steam cleaning for 15 seconds before application of veneering ceramic. The samples were divided into three groups of 40 samples each and each group was layered with a different veneering ceramic (IPS e.max Ceram, Vita VM9, or GC Initial Zr-FS) The groups were further divided into four subgroups of 10 samples in order to apply four different cooling processes.

Three different veneering ceramics were applied to the groups according to manufacturers' instructions by using a layering technique. A specially designed stainless-steel mold was used to standardize the veneering ceramic size for all samples. A zirconia disc-shaped specimen was placed in this mold, with a space of 10 mm in diameter and 3 mm in height above the zirconia material to condense the veneering ceramic.

Table 3. Technical data on the Kavo Everest ZS

Kavo Everest ZS	
ZrO ₂	99,60%
Y ₂ O ₃	5,00%
Al ₂ O ₃	0,25%
Other metal oxides	< % 0,15
Average grain size	0,52 ± 0,05 µm
Coefficient of Thermal Expansion (CTE)	10,0 x 10 ⁻⁶ K ⁻¹
Thermal Conductivity	2,5 W/mK
Chemical Solubility	10 µg/cm ³
Flexural Strength	1200 MPa
Elasticity Modulus	210 GPa
Fracture Toughness	8 MN/m ^{1/2}

Firing process

The Austromat D4 (Dekema Dental, Freilassing, Germany) porcelain furnace was used for the slow cooling process because of the accuracy of control of this furnace in bringing the samples temperature down through the glass transformation temperature range. The firing table mechanism allows the table to exit the furnace in a vertical downwards direction. After the first dentin firing schedule, four different cooling rates; maximum, 25°C/minute, 50°C/minute and 75°C/minute through the glass transformation temperature range of veneering ceramics were used. Maximum cooling consists of immediately bringing the samples temperature down in the furnace after the end of the firing schedule. Then all samples were exposed to ambient air (~23°C).

Shear Bond Strength Test

Shear bond strengths (SBS) were determined according to ISO TR 11405, using a universal testing machine (Shimadzu AG-IS, Shimadzu, Kyoto, Japan) at a crosshead speed of 0.5 mm/min. The obtained load values were converted into the megapascals (MPa) by dividing the failure load (N) by the bonding area (mm²).

Microstructural analysis

Microstructural characterization was conducted using scanning electron microscopy (SEM) and X-ray diffraction (XRD) analysis. Cross-sections of the samples were evaluated using SEM (JSM-7000F, Jeol, Tokyo, Japan) at ×500 magnification to observe their crack pattern and grain size.

Representative samples that showed, possible occurrence of a t-m transformation of zirconia during the firing process of the veneering ceramic were detected by using x-ray diffraction (XRD). XRD patterns were collected using a diffractometer (X'Pert Pro, PANalytical, The Netherlands) within the 2θ range of 25–65, covering the positions of the highest peaks of tetragonal and monoclinic phases of ZrO₂. CuKα radiation was generated at 40 kV and 45 mA. Mass fraction of monoclinic phase (X_m) was calculated using the methods of Garvie-Nicholson (13) and Toraya et al (14).

Statistical analysis

Data were analyzed with IBM SPSS V23 (IBM SPSS, Armonk, NY, USA). Two-way analysis of variance was used to compare mean values according to veneering ceramics and cooling process. For the comparison of the main effects, the Bonferroni correction and paired comparisons were employed. Analysis results are presented as mean and standard deviation. The significance level was set to p < 0.05.

Results

Shear bond strength

The main effects of veneering ceramics on shear bond strength were found to be significant (p = 0.042). Average shear bond strength value was 25.14 ± 7.55 MPa (Min 13.49 MPa - Max 44.01 MPa) in the Vita VM9 group, 24.14 ± 7.78

MPa (Min 10.58 MPa - Max 49.44 MPa) in the IPS e.max Ceram group and 21.12 ± 7.86 MPa (Min 10.95 MPa - Max 36.40 MPa) in the GC Initial ZR-FS group. Mean shear strength values differ according to the cooling process (p < 0.001). While the mean shear bond strength value was 19.68 ± 6.08 MPa (Min 10.58 MPa - Max 29.83 MPa) in Max-groups, it was obtained as 22.27 ± 5.74 MPa (Min 11,34 MPa - Max 31,96 MPa) in "75" groups, 23,88 ± 7,75 MPa (Min 11,34 MPa - Max 39,09 MPa) in "50" groups and 28,04 ± 9,22 MPa (Min 14,14 MPa - Max 49,44 MPa) in "25" groups. The average value obtained in the "25" groups is higher than "Max" and "75" groups. The value obtained in "50" groups does not differ from other groups (Table 4 and 5) (Figure 1).

Veneering ceramic and cooling process interaction did not have a significant effect on the average value (p = 0.938). The measurements obtained at different cooling process in Vita VM9 did not differ from the measurements in IPS e.max Ceram and GC Initial ZR-FS. The main effect of the cooling process on shear bonding strength was higher than the main effect on the veneering ceramic. While the partial eta squared value obtained for the cooling process was 0.160, the value obtained for veneering ceramics was 0.057 (Table 5).

Microstructural analysis

The monoclinic phase ratio increased in groups in which the fast-cooling process was applied (Figure 2). Therefore, an increase in monoclinic volume was accompanied with a decrease in mechanical properties. SEM photographs revealed that among the three different veneering porcelains, they

Table 4. Descriptive statistics and multiple comparison results for shear bond strength according to veneering ceramic and cooling process

	Vita VM9	IPS e.max Ceram	GC Initial ZR-FS	Sum
Max	20,62±5,73 MPa	20,34±6,37 MPa	18,09±6,42 MPa	19,68±6,08 ^A MPa
75	23,84±4,42 MPa	23,06±4,79 MPa	19,91±7,36 MPa	22,27±5,74 ^A MPa
50	25,32±8,45 MPa	26,10±7,09 MPa	20,21±7,02 MPa	23,88±7,75 ^{AB} MPa
25	30,79±7,93 MPa	27,08±10,82 MPa	26,27±9,01 MPa	28,04±9,22 ^B MPa
Sum	25,14±7,55 ^a MPa	24,14±7,78 ^{ab} MPa	21,12±7,86 ^b MPa	

^{a-b} There were no statistically difference between veneering ceramics with the same letter; ^{A-B} There were no statistically difference between cooling process with the same letter.

Table 5. Effects of veneering ceramic and cooling process on shear bond strength

	F	p	Partial Eta Squared
Veneering ceramic	3,267	0,042	0,057
Cooling process	6,878	<0,001	0,160
Veneering ceramic * Cooling process	0,296	0,938	0,016

had similar microstructural appearances as a function of the cooling process. The mode of failure analysis results for the groups showed mixed failure mode with both cohesive failure within the veneering ceramic and adhesive failure with the veneering ceramic debonding from zirconia. A direct view of the fracture surface at the zirconia framework showed that a thin layer of the veneering ceramic remained on the surface with exposure of the zirconia structure in groups in which the fast-cooling process was applied (Figure 3).

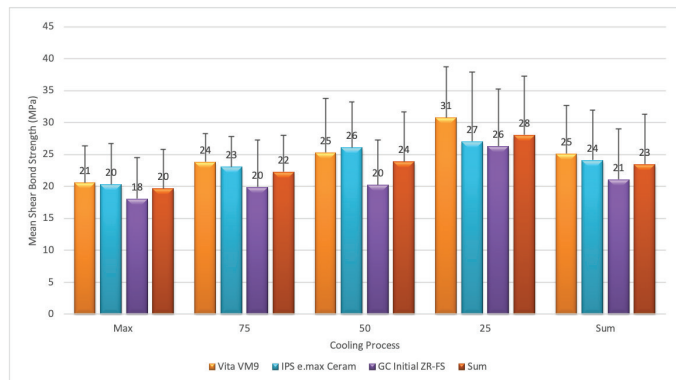


Figure 1. Mean shear bond strength results of the groups.

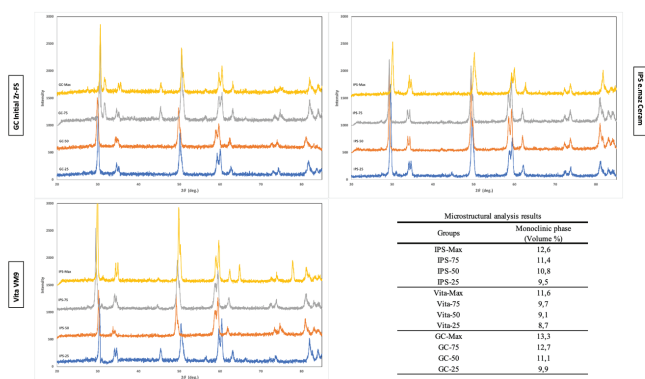


Figure 2. XRD patterns and volume fraction (V_m) results of the monoclinic phase of the groups.

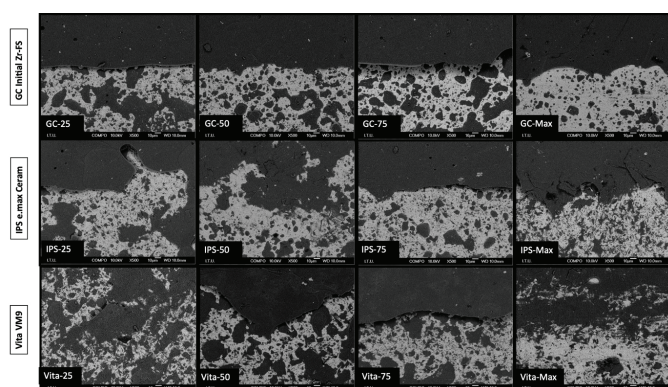


Figure 3. SEM photographs of the groups.

Discussion

Numerous factors have been reported to cause chipping failure in zirconia-based ceramics. Chemical bonding, mechanical bonding provided by surface roughness, the type and amount of defects at the interface, the wetting property of the veneering ceramic and the difference in the thermal expansion coefficient between zirconia and the veneering

ceramic are among the factors affecting the bond strength. In addition, the thickness ratio of zirconia-veneering ceramics, the geometry of the restoration and inadequate veneering ceramic design, the magnitude of the applied force, the direction-frequency and the location of the occlusal forces, the elasticity modulus and conductivity differences of the framework and veneering ceramics, and the thermal changes observed during the firing of the veneering ceramics were indicated as other failure factors (6-9,15,16). Among these factors, the effect of the thermal changes seen during the firing of the veneering ceramics, especially the cooling process, on the bond strength between zirconia and veneering ceramics is not clear (17,18). Since the manufacturers producing veneering ceramics compatible with zirconia suggest applying a slow cooling process after firing, our research aimed to examine the effect of the thermal changes observed during the firing of ceramics applied on zirconia on the bond strength.

Previous studies investigated different cooling processes (18-20). In the present one, four different cooling processes were performed after the application of the veneering ceramics. The cooling process was applied in a similar way to the firing (similar to the heating process) applied by Tan *et al.* (21). It was stated that the samples that underwent slow heating and cooling showed higher fracture strength, but the results were not statistically significant. The failure occurred cohesively in ceramics in the region close to the zirconia framework (21). In another study, it was concluded that it was very difficult to analyze the results due to fracture patterns and factors affecting residual stress since there was no standardization regarding the cooling process and sample design (18). In our experiment, in groups that were subjected to the fastest cooling process, the samples were taken out of the furnace as soon as the firing process was completed, and the cooling process was initiated. It has been noted that this process could allow for a comparison which is similar to the steps a dental technician follows to remove restorations from the furnace immediately after firing (20).

The cooling process was performed in the furnace up to the glass transition temperature of the ceramic. The long-term cooling process is used to prevent residual stress formation in glasses and materials containing a glass matrix (22). At an annealing point above 50°C, the glass is hard enough not to deform and soft enough to relieve stresses (22-24). Therefore, the heat of glass transition had a significant effect on the magnitude of the residual stress in the ceramic and it was considerably affected by the cooling process (23,24). Above the glass transition temperature, the ceramic could make molecular movements that do not cause any stress and behave as a viscous structure that allows reshaping (8,23,25). As the temperature decreases, the viscosity of the ceramic increases and the molecular displacement becomes more difficult with the amount of thermal energy. The ceramic transforms into the elastic solid phase below the glass transition temperature (where there are no structural changes and stress accumulation may occur) (8,23).

The properties of the liquid vary, depending on the time during the transition process to the glass phase, and slow cooling should have been applied up to the glass transition temperature in order to prevent stress due to thermal transitions in ceramic and glass-containing materials. Thus, a ho-

mogeneous heat dissipation could be achieved between the inner and outer layers of the ceramic (26). For this reason, it has been concluded that the cooling process up to the glass transition temperature is more important in the formation of stress, and in our study, it was decided to perform the cooling process up to this temperature in the furnace in a controlled manner.

The stress in the veneering ceramics is an important factor in determining the longevity of the restoration. The thermal incompatibility between the framework and the veneering ceramics creates compressive or tensile stresses in the veneering ceramics, depending on whether the thermal expansion coefficient of the veneering ceramics was lower or higher than the framework. Ceramics were resistant to compressive stresses and but not to the tensile ones, and the occurrence of compressive stress in the veneering ceramic is a desirable situation, because in this way the veneering ceramic is strengthened, and the fracture resistance increases. Due to the fact that the thermal expansion coefficient of the veneering ceramic was somewhat lower than or close to that of the framework, the favorable compressive stresses may occur during the cooling process (27). When the thermal expansion coefficient of the veneering material is higher than that of the framework material, delamination and micro cracks could be observed in the veneering. In metal-ceramic systems, the excessive stresses caused by the mismatch of the thermal expansion coefficient could be partially compensated by the elastic or plastic deformation in the metal (28). However, zirconia has a lower thermal expansion coefficient than other ceramics. More destructive stresses occur in veneering ceramic in zirconia-based restorations. Therefore, it was previously reported that veneering ceramic resistance was an important parameter in long-term success (29). For this reason, in recent years, special veneering ceramics have been developed with the same or lower thermal expansion coefficient than zirconia. In our study, a zirconia framework and veneering ceramics with close and compatible thermal expansion coefficients were used.

Tempering is a method created by heating the glass to a temperature slightly below its softening degree and suddenly cooling it to room temperature and used to strengthen the ceramic (30). While this process is recommended for metal-based ceramic systems, the recommendations for veneering ceramics applied on a zirconia framework are different. Post-firing slow cooling (with the furnace closed from liquid phase to glass transition temperature) has been recommended for some ceramics (31). Manufacturers are currently concentrating their efforts on reducing the stress areas and cracks in ceramics. Previous research on this subject indicated that the fast cooling rate after firing might cause a high thermal tempering effect and residual stresses on the surface (10,32). Another factor in the residual stress formation that causes fractures in the veneering ceramic is related to the fact that zirconia is a weak conductor compared to metal alloys and even to other full ceramic frameworks. When fast cooling is applied to metal-based ceramics, veneering ceramics can cool rapidly from the surface to the inside because metal is a good conductor, but fast cooling cannot occur in zirconia-based ceramics. Veneering ceramic's surface hardens after fast cooling, but the inner part adjacent to the zirconia remains above the glass transition

temperature for a long time, and wide thermal differences occur between the inner and outer layers (25,33). Benetti *et al.* (34) found that the temperature difference between the inner and outer layers was lower in the samples with slow heating and cooling compared to the samples with fast heating and cooling. In addition, the temperature difference between the layers in the metal-ceramic samples with fast cooling was found to be lower than that of the zirconia-ceramic samples. This result is thought to be due to metal being a better conductor than zirconia.

During the application of the veneering ceramic, the zirconia framework is exposed to existing humidity and increasing heat, and the zirconia is not stable due to the spontaneous t - m phase transformation. This can cause a decrease in mechanical properties. Heat, humidity, particle size, micro and macro cracks in the material can affect the t - m phase transformation of the stabilizing oxide type and amount. The most critical temperature range for this conversion is 200-300 °C and the conversion is increased in the presence of water or steam. However, it has been stated that the effects of degradation at low temperatures on Y-TZP can only be significant after years (35,36). Tholey *et al.* detected monoclinic crystals at the zirconia-veneering ceramics interface using the SEM and XRD method (37,38). In zirconia, a volume increase of ~ 4% occurs with the monoclinic phase formed due to phase transformation. This increase in volume may develop due to the increase in humidity and temperature in the environment during the firing of the veneering ceramics (38). It was reported in another study that, the decrease in strength may depend on the amount of the structure being transformed, and there may be a significant reduction in strength when transformation occurs in a large part of the structure (39).

In the present study, the firing procedure (fast cooling after firing) decreased the shear bond strength of Vita, IPS and GC samples. XRD analysis revealed an increase in the monoclinic phase volume ratio on the surface which was observed in the groups with fast cooling after firing (in the maximum groups) compared to the groups in which slow cooling was applied (in the 25 groups). The volume increase of the monoclinic phase was also observed in the 75 and 50 group samples, and it was determined that the highest monoclinic phase volume ratio was in the GC maximum group (13.3%), and the lowest monoclinic phase volume ratio was in the Vita 25 group (8.7%). In general, it was observed that as the cooling rate increased in all groups, higher monoclinic phase volume ratios occurred. In addition, lower bond strengths were observed in the samples that underwent fast cooling (in the maximum groups). The highest monoclinic phase volume ratio was found on the surface of the GC maximum group with the lowest bond strength value, and the lowest monoclinic phase volume ratio was seen on the surface of the Vita 25 group with the highest bond strength. Taken together, it can be stated that the shear bond strength was also found to be lower in the samples with a higher monoclinic phase volume ratio. The fast-cooling process applied after the firing process may trigger the transformation of the metastable tetragonal phase into the monoclinic phase and could have weakened the structure. When the samples are evaluated in terms of failure type; combined fractures (cohesive failure in the veneering ceramic and adhesive failure at the inter-

face) were detected. In the SEM analysis, the zirconia framework in GC max. group was more exposed at the interface, although the fracture started in the veneering ceramic. This finding may support the low shear bond strength value.

There may be defects in ceramic material due to manufacturing process. Internal defects, grain size mismatches, large grain size, regions with different phases are microcracks due to the expansion and contraction of different crystal phases. External defects, on the other hand, occur after manufacturing processes and can be caused by voids, processing damage, sediments and foreign material residues. The superficial defects are always present in ceramics, and volumetric defects can re-surface with abrasion as well as during the polishing process, which could further weaken the structure (40). Despite the maximum care and attention in our study, the inherent structural defects might also have caused failure. The limitations of our study include the use of samples with different anatomical geometries to mimic the clinical situation and the ability to create fatigue by using a chewing simulator.

Conclusion

Within the limitation of this experimental study, it could be stated that the thermal changes during the firing process of veneering porcelain over a zirconia framework influenced the bond strength of the all-ceramic bilayered system. The fast cooling process reduced the strength of the bond between the zirconia framework and veneering ceramics. Slow cooling, especially after veneering firing, may reduce the bond failure, and therefore, it may prolong the longevity of the ceramic restoration.

Türkçe özet: Zirkonya altyapı üzerine uygulanan seramiklerin ısısal değişimlerinin bağlantı dayanımlarına etkisinin incelenmesi. Amaç: Bu çalışmanın amacı; zirkonya altyapılar üzerine uygulanan seramiklerin fırınlanması sırasında görülen ısısal değişimlerin bağlantı dayanımlarına etkisinin incelenmesidir. Gereç ve Yöntem: Çalışmamızda single yttria stabilize zirkonya polikristali (Y-TZP) altyapı seramiği (Kavo Dental GmbH) ve 3 farklı üstyapı seramiği (IPS e.max Ceram, Vita VM9 ve GC Initial Zr-FS) kullanıldı. Zirkonya bloklardan CAD/CAM sistemi (Kavo Everest) kullanılarak yüz yirmi adet standart disk şeklinde örnekler hazırlandı. Üstyapı seramiklerine fırınlama sonrası dört farklı soğutma işlemi (maksimum, 25°C/dk, 50°C/dk ve 75°C/dk) uygulandı. Örneklere Makaslama Bağlantı Dayanımı Testi (SBS) uygulandı. Seramiklerin yüzeyi Taramalı Elektron Mikroskopu (SEM) kullanılarak incelendi. Zirkonyadaki t-m faz dönüşümü, X-Işını Difraksiyon Analizi (XRD) ile değerlendirildi. Çalışma istatistiksel olarak, iki yönlü varyans analizi, Bonferroni düzeltmesi ve ikili karşılaştırmalar kullanılarak incelendi. Bulgular: Üstyapı seramiklerinin, makaslama bağlantı dayanımı üzerindeki ana etkilerinin anlamlı olduğu tespit edilmiştir (p=0.042). Ortalama makaslama bağlantı dayanımı değerleri soğutma işlemine göre anlamlı farklılık göstermiştir (p<0,001). Hızlı soğutma işlemi uygulanan gruplarda monoklinik faz oranında artış gözlemlenmiştir. Sonuç: Zirkonya altyapı üzerine uygulanan seramiklerin, fırınlama işlemleri sırasındaki ortaya çıkan ısısal değişiklikler, çift tabakalı seramik sistemin makaslama bağlantı dayanımını etkilemiştir. Yavaş soğutma işleminin, çift tabakalı seramik örnekler için daha yüksek bağlantı dayanımı sağladığı tespit edilmiştir. Anahtar Kelimeler: Zirkonya, Üstyapı seramiği, CAD/CAM, Bağlantı dayanımı, Soğutma işlemi

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